Sources and Spatial Variation of Dissolved Organic Matter in Luoma Lake

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ABSTRACT

In order to investigate the characteristics, sources and variations in the composition of dissolved organic matter (DOM) during the wet and dry seasons in Luoma Lake, UV-vis and 3D-EEMs techniques combined with PARAFAC were used to analyse the changes in DOM content and components in dry and wet season. The results of the study showed that PARAFAC identified three types of fluorescent fractions, C1 (humus-like fraction), C2 (fulvic-like fraction) and C3 (protein-like fraction). The fluorescence intensity of the DOM fraction was significantly higher in wet seasons than in dry seasons. The spectral parameters of DOM indicated that the water body of Luoma Lake was mainly autogenous due to the combined effects of endogenous and exogenous inputs. The relative concentrations of DOM in Luoma Lake were lower in the dry season than in the wet season, and the proportion of hydrophobic components and the degree of aromatization of DOM were higher in the wet season. The findings demonstrated that hydrological seasonality and anthropogenic activities were key drivers for the DOM compositions and sources variations, which will improve our understanding on the crucial role of DOM in biogeochemical cycle, as well as help to guide water quality protection in Luoma Lake.

Keywords: Luoma Lake, Dissolved organic matter (DOM), parallel factor analysis (PARAFAC), Hydrological season

1. Introduction

Luoma Lake is a typical shallow freshwater lake located in the downstream of the Yimusi River network area of the Eastern China (34°00′–34°14′N, 118°06′–118°18′E). The lake plays an important role as the key storage reservoir and hydro-conveyance transfer station for the river network. It is also the centralized public drinking water source for millions of people in the cities of Suqian and Xinyi and the wetland reserve in Jiangsu Province. The water quality of the lake is crucial to the safety of the drinking water for the local public, pollutant behavior in the river network system, and security of the regional ecological environment. As it is encompassed by many villages and factories, Luoma Lake directly or indirectly receives a large amount of tailwater from domestic and industrial sewage treatment via tributaries and some non-point wastewater sources every year (Liu et al. 2017; Yan et al. 2017; Xing et al. 2018). Therefore, it is necessary to study the water quality changes of the lake environment.

Dissolved Organic Matter (DOM), primarily composed of humic substances, carbohydrates, and proteins, plays a crucial role in processes such as heavy metal mobility, nutrient cycling (nitrogen and phosphorus), and the carbon cycle (Zhang et al. 2010; Schmidt et al. 2017). DOM is widely present in surface water and groundwater as a consequence of different hydrological, biological, and geological interactions (Leenheer & Croue 2003; Mayayorga et al. 2005). It supports heterotrophic microbial metabolism (Stedmon et al. 2011) and, to some extent, affects the integrity and function of riverine ecosystems. DOM in lake can be derived from autochthonous inputs (e.g., phytoplankton, aquatic plants, and heterotrophs), allochthonous inputs (e.g., soil leaching, rock weathering, and atmospheric deposition), and anthropogenic inputs (e.g., industry, wastewater, intensive agriculture, and farms) (Fisher et al. 2004; Elliott et al. 2006; Giorgio & Pace 2008; Griffith & Raymond 2010; Fashing et al. 2014). Many researchers have made achievements in semi-quantitative and qualitative research on DOM using three-dimensional fluorescence combined with parallel factor analysis (EEMs-PARAFAC). Shang et al. (2019) emphasized the importance of DOM in the study of DOM intakes in water bodies, and strengthened the relationship between nutrient index and DOM. Hu et al. (2017) applied parallel factor analysis to analyze the source and composition characteristics of DOM in Hongze Lake, revealing its water quality status and influencing factors. Therefore, the source, characteristics and other information of DOM can be judged according to different components identified by the water body DOM. Accordingly, these previous studies have demonstrated the effectiveness of EEMs–PARAFAC in the analysis and identification of DOM sources and its compositional characteristics.

In this study, the temporal and spatial distribution characteristics of DOM in Luoma Lake under different hydrological scenarios were analyzed, and the source and composition characteristics of DOM were discussed. The results can provide useful information to guide source control, sewage interception, and water environmental quality improvement in the Luoma Lake.

2. Materials and methods

2.1 Sampling design and collection

Luoma Lake is located in the Yimusi River network area of the northern Jiangsu Plain, and is surrounded by Suqian city and Xinyi city (Fig. 1). It has an average water depth of 3.3 m and an average surface water area of 260 km^2 (Liu et al. 2017; Xing et al. 2018). There are four main lacustrine rivers directly connected to Luoma Lake, namely the Yi River, Huangdunhu River, Xinyi River, and Pihong River. The local public drinking water source areas of Suqian and Xinyi are located in the estuaries of the Pihong River and Xinyi River, respectively. The northern and southern lake districts contain the important provincial wetland reserves of Luoma Lake (Suyu District) and the abandoned Yellow River (Suyu District), respectively.

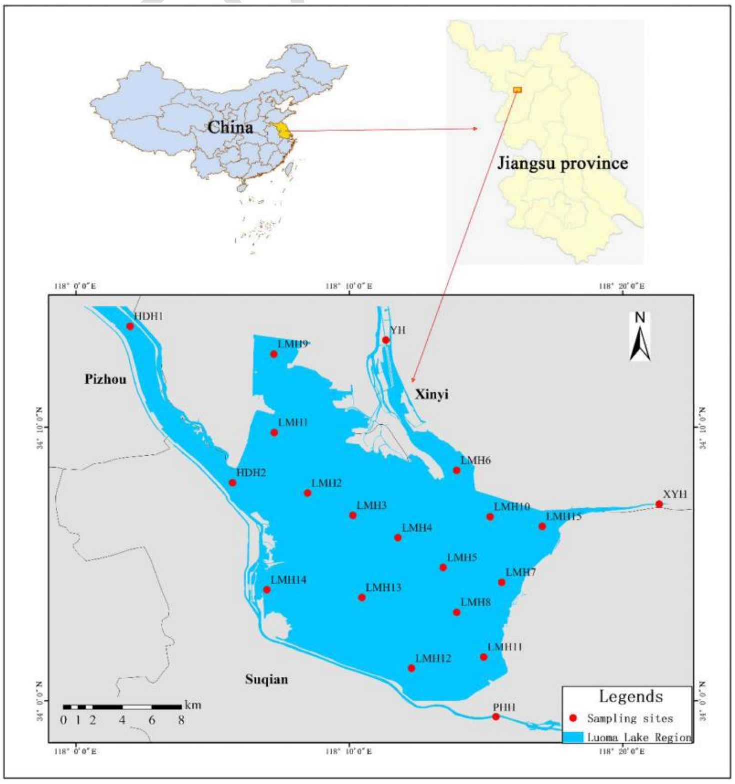

Figure 1. Study area and locations of sampling sites in Poyang Lake and Lake Taihu, China.

In this study, a high-resolution river-estuary-lake monitoring network with 21 sampling sites was established, which included 4 sites located in lacustrine rivers (YH, HDH1, XYH, and PHH), 4 sites located in estuaries (HDH2, LMH15, LMH16, and LMH6), and 13 sites located in the lake region (LMH1, LMH2, LMH3, LMH4, LMH5, LMH7, LMH8, LMH9, LMH10, LMH11, LMH12, LMH13, and LMH14), which covered the environmentally sensitive areas of public drinking water sources and important wetland reserves in Luoma Lake. The field sampling of water (0-1 m) from these monitoring sites was conducted in April (dry season), July (wet season) 2024. A polypropylene (PP) bucket was used to collect 6 L of surface water per site, which was stored in a brown PP bottle pre-rinsed with Milli-Q water. All samples were transported to the laboratory in ice boxes, where the water samples were stored at 4 °C in the dark until further analysis.

2.2 DOM characterization

The TOC concentration in water samples was measured using a TOC analyzer (TOC-L CPH, Shimadzu). Absorption spectra were measured using a UV–Vis spectrophotometer (Cary5000, Varian). A series of absorption indices, including a_{254}、$SUVA_{254}$ and S_R were computed according to published methods (Weishaar et al. 2003; Zhang et al. 2021). Fluorescence EEMs of water samples were scanned using the Horiba FluoroMax-4 fluorescence spectrometer with a 1 cm quartz cuvette. The excitation wavelength (E_x) was set at 200－500 nm in steps of 5 nm. The emission wavelength (E_m) ranged from 250 to 600 nm with a 1 nm interval and a scanning speed of 1200 nm/min. Water Raman scatter peaks and Rayleigh scatter peaks were eliminated based on the corrected methods of previous studies (Stedmon & Bro 2008; Li et al. 2021). The EEM results were normalized as Raman units (R.U.) using the integrated area of the Raman peak of daily measured Milli-Q water excited at 350 nm (Lawaetz & Stedmon 2009). Various fluorescence indices, such as fluorescence index (FI), biological index (BIX), and humification index (HIX), were computed following the published methods (Hansen et al., 2016).

$$FI = \frac{F(E\text{x} = 370\text{nm}, Em = 470nm)}{F(E\text{x} = 370\text{nm}, Em = 520nm)}$$

$$BIX = \frac{F(E\text{x} = 310\text{nm}, Em = 380nm)}{F(E\text{x} = 310\text{nm}, Em = 430nm)}$$

$$HIX = \frac{F(Ex = 255\text{nm}, Em = 435 \sim 480nm)}{F(Ex = 255\text{nm}, Em = 300 \sim 345nm)}$$

2.3 Data analysis

The sampling point distribution map was drawn using ArcGIS 10.2 (Environment System Research Institute Inc., Redlands, CA, USA). Data processing and correlation analysis were performed using Origin 2022 (study version; OriginLab Corp., Northampton, MA, USA) and Excel 2016 (Microsoft Corp., Redmond, WA, USA). PARAFAC analysis of EEMs was carried out using the DOMFluor toolbox in MATLAB 2018b (MATLAB, MathWorks Inc., Natick, MA, USA), and the results were subjected to cluster analysis using SPSS 26.0 (IBM Corp., Armonk, NY, USA).

3. RESULTS AND DISCUSSION

3.1 Absorption spectral characteristics of the DOM

To acquire more details about the chemical characteristics of DOM, UV-Vis spectrophotometry was applied in our study and the relevant parameters are shown in Fig.2-3. Generally, TOC concentration and DOM absorption a_{254} in the wet season is higher than that in the dry season. And the TOC concentration gradually decreases from the northern area into the lake to the southern open water area. This indicates that the input of organic matter was higher in the wet season, and the source of DOM is mainly terrestrial. There was no significant difference in $SUVA_{254}$ and S_R between wet season and dry season. In both wet and dry seasons, $SUVA_{254}$ gradually decreases from the northwest into the southeast open area, it is particularly significant indicating that the DOM in Luoma Lake is mainly river input.

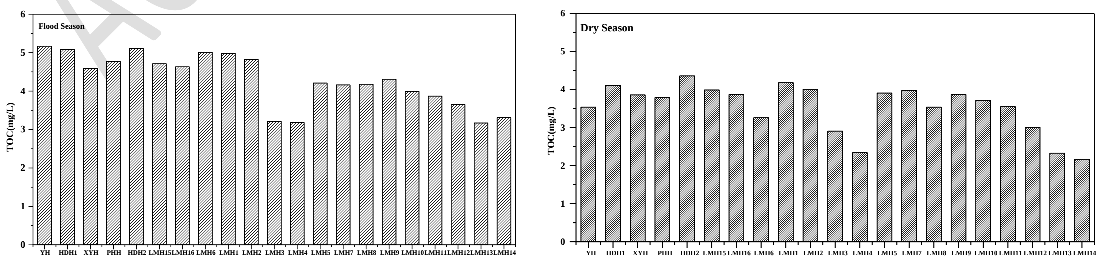

Figure 2. Distribution of TOC concentrations in Luoma Lake

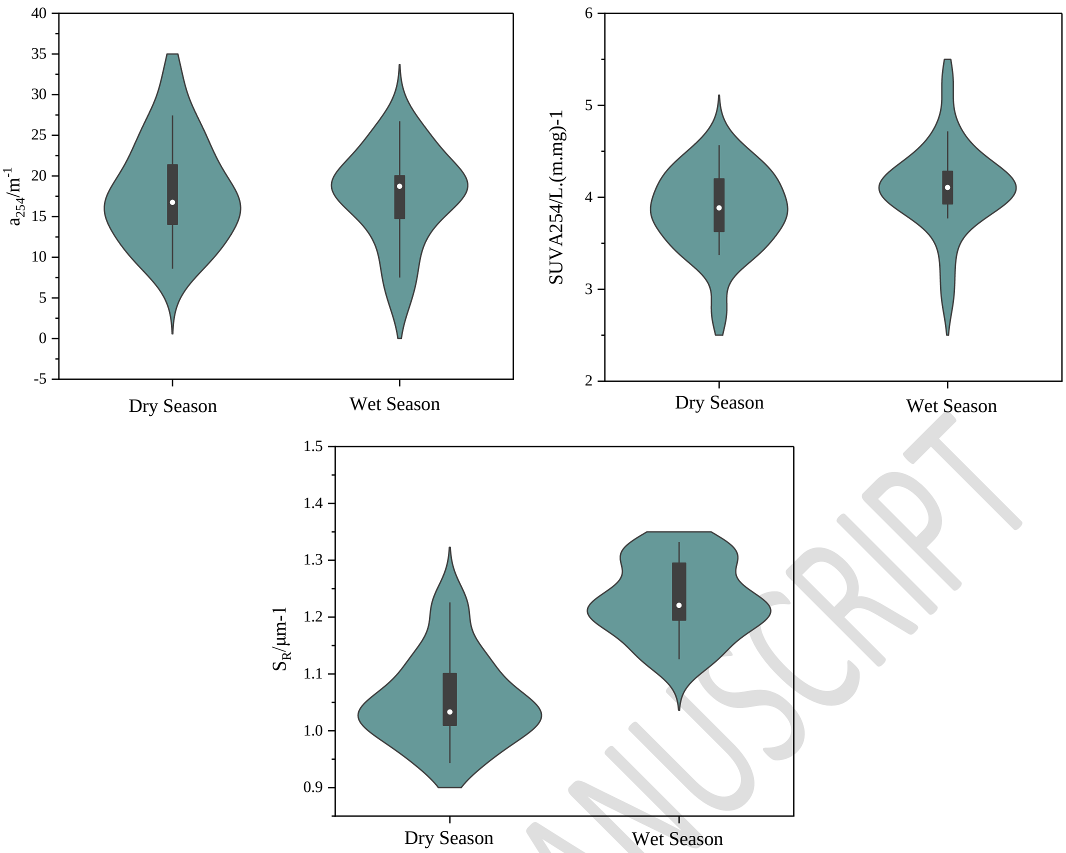

Figure 3. **a_{254}、$SUVA_{254}$ and S_R of DOM in in Luoma Lake**

In wet season, the $SUVA_{254}$ values vary from 2.93 to 5.20. Compared with other sampling points, the $SUVA_{254}$ values at points HDH2 and LMH1 are remarkably larger, and the higher degrees of aromatization and humification are due to the strong influence of human activities. The S_R values range from 1.13 to 1.33, are similar to those of Songhe River (0.89±0.11), Pingqiao River (0.90±0.11) and Zhongtian River (0.96±0.10). Studies have shown that when DOM stays in reservoirs and lakes for a long time, photobleaching can transform large molecules of DOM into small molecules in water (Catalán et al. 2016; Song et al. 2019). In Luoma Lake, the water in rivers has a short residence time and weak organic degradation, resulting in a larger molecular weight of DOM detected. In our study, there was no evident relationship between $SUVA_{254}$ and S_R. Similar finding was reported for the DOM in the Three Gorges Reservoir (Jiang et al. 2018). These findings suggest that the relationship between $SUVA_{254}$ and S_R is not always linear, the aromaticity of DOM reduced with inputing urban wastewater, and having a lesser impact on molecular weight.

3.2. Three-dimensional fluorescence characteristics of the DOM

3.2.1. Fluorescent component characteristics

The composition of the DOM fluorescence in Luoma Lake were analyzed by PARAFAC analysis. In total, three components of two types were identified in the basin and its tributaries. Comparison of component type with those reported by Coble et al. (1990) revealed that they are humic-like (C1, C2) and protein-like (C3) components.

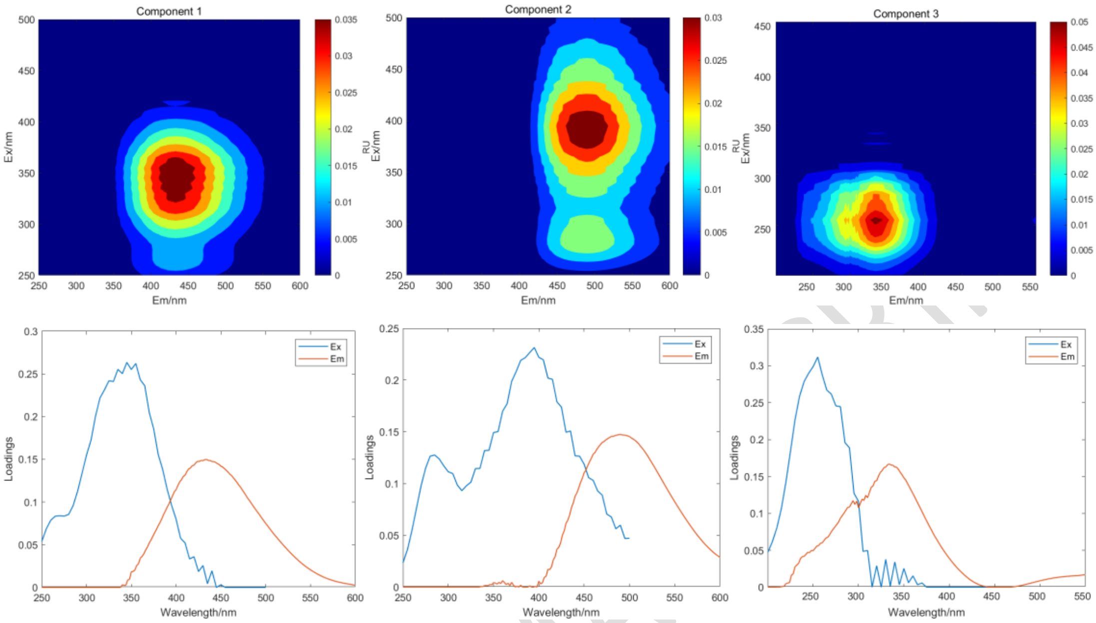

Figure 4. Fluorescence spectra of the three PARAFAC components and the three-component-model was well validated using split-half validation

C1 340/448 nm match fluorescence peak F, which resembles the pattern of a typical fulvic acid-like peak. Such components originate primarily from domestic sewage and represent a typical terrestrial fulvic-like substance (Garcia et al. 2018). C2 280(390)/490 nm resembles the pattern of a typical humic acid-like peak. C3 (250/340 nm) correspond to fluorescence peak T, which mirrors the pattern of a typical tryptophan-like peak. Such components are prone to biodegradation, strongly influenced by human activities (Yi et al. 2017), and derived mainly from urban domestic sewage and food industry wastewater (Francisco et al. 2020).

DOM sources in natural water mainly include exogenous sources (such as terrestrial and anthropogenic sources) and endogenous sources (such as microbial metabolism and extracellular secretion) (Lawaetz et al. 2009). In natural water, DOM components are usually humus type (Cory et al. 2005), while protein-like components in water affected by microbial metabolism and human activities have a higher content. In order to clarify the relative contribution of DOM components, the fluorescence intensity data of each component were analyzed and calculated. On the whole, the fluorescence intensity of DOM in wet season was significantly higher than that in dry season. Organic matter in surface water is mainly derived from endogenous generation and terrigenous transport. The average water temperature of surface water was 16.5 ℃ in dry period and 28.3 ℃ in wet period. The rising water temperature promoted the microbial activity in water (Zhang et al. 2010), accelerated the decomposition of organic matter in water and the release of organic matter in sediment, and was one of the reasons for the increase of DOM fluorescence intensity.

The fluorescence intensity of C1 and C2 components in Luoma Lake during the wet season was significantly higher than that during the dry season ($p<0.01$). Overall, the three components show a decreasing trend from north to south under different hydrological scenarios, the spatial distribution of fluorescence intensity of components C1 and C2 during the wet season is similar, with higher fluorescence intensity in the northern and central lake areas, and then decreased along the water flow. At the same time, during the wet season, the fluorescence intensity of C3 components increased significantly at sample site HDH2、LMH15、LMH16 and LMH6, which was due to the sample sites near to the bank. Abundant runoff during the wet season carried protein-like components produced in the process of self-biogenesis of terrestrial plants and soil organic matter and the protein-like components produced by domestic sewage discharge.

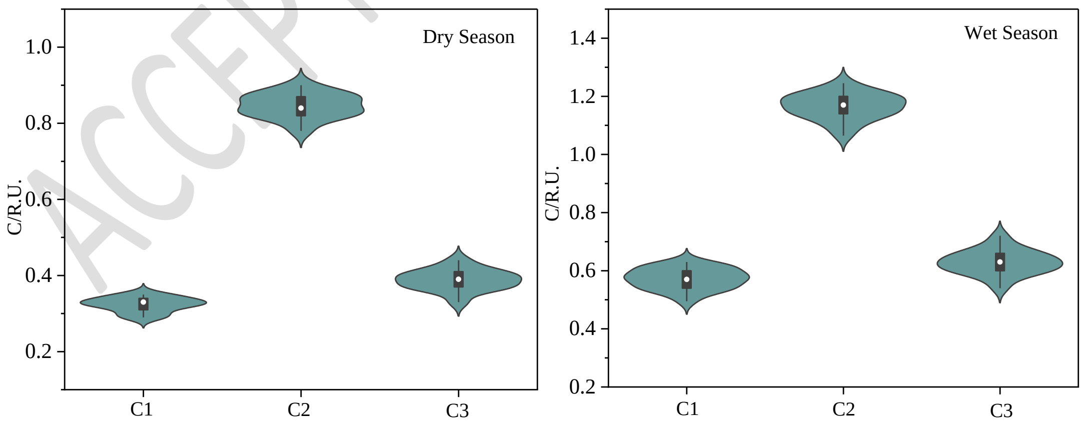

Figure 5. Properties of the three components (C1、C2、C3) during the flood season and the dry season

The percentages of fluorescent components (C1, C2, C3) in the dry and wet seasons are summarized in Fig. 6. The percentage of three fluorescent components demonstrated significant differences between two seasons ($p < 0.001$). The percentage of humic-like (C1) was 55% in the wet season, 1.57-fold higher than that in the dry season. The percentage of protein-like (C3) was 28% in the dry season, 1.65-fold higher than that in the wet season.

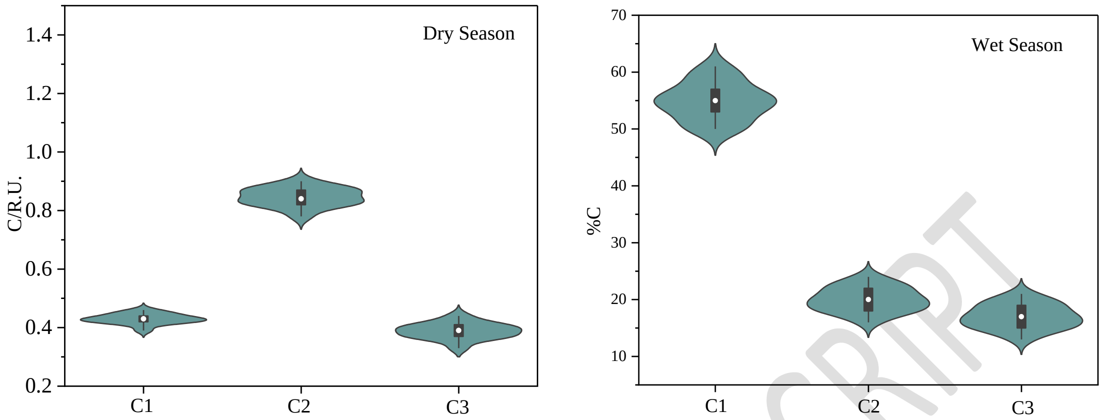

Figure 6. The percentage of three components (C1、C2、C3) during the flood season and the dry season

Kramer & Herndl (2004) have demonstrated that humic-like substances are less susceptible to microbial degradation than protein-like substances in water bodies. As reported by Hudson et al. (2007), humic-like substances account for a relatively large proportion of DOM in natural water bodies, whereas protein-like peaks are enhanced by human activities. The large proportions of humic-like substance (C1, C2) for the DOM in Luoma Lake are consistent with the results of Gan (2013). In contrast, the low proportion of protein-like substance (C3) is associated with the dominant terrestrial inputs of DOM pollutants in water across the sampling points. The higher percentage of C1 and C2 (Fig. 6) indicated a higher percentage of humic-like in the wet season than in the dry season. This was consistent with the observation obtained from a254. The larger percentages of C3 indicated a higher percentage of protein like in the wet season than in the dry season. The seasonal variations of component percentages were also consistent with the previous studies (Zhao et al., 2016), suggesting that DOM was primarily contributable to humic-like substance in the wet season, while protein-like in the dry season. The seasonal variation of DOM fluorescence demonstrated variable dominant sources of DOM in difference seasons, with terrestrial inputs dominant in the wet season, and autochthonous sources in the dry season.

3.2.2. Fluorescence spectral parameters

To identify the source of DOM in the water bodies, the fluorescence parameters of the Luoma Lake were plotted (Fig.7). Fluorescence index (FI) is used to characterize the source of the DOM (Yang et al. 2019). When FI > 1.90, DOM is primarily origin from microbial , while FI < 1.40 DOM is basically from autochthonous inputs. In Luoma lake, the FI values of the dry season samples ranged from 1.28-1.98, with a mean of 1.66; the FI values of the wet season samples ranged from 1.04-1.45, with a mean of 1.26. The mean FI values of the samples from different water periods were all within the range of 1.2-1.8, indicating that the DOM in Luoma Lake has both autochthonous and allochthonous sources. The autochthonous DOM mainly comes from the release of sediments at the bottom of the lake and microbial metabolism, while the allochthonous DOM is produced by runoff or human activities. The mean FI value of the wet season samples is significantly lower than that of the dry season samples (P ＜0.05), indicating that the proportion of allochthonous input in the DOM component of the wet season is increased, which is consistent with the fact that the wet season has more precipitation and the reservoir runoff increases, and the external input contribution is high.

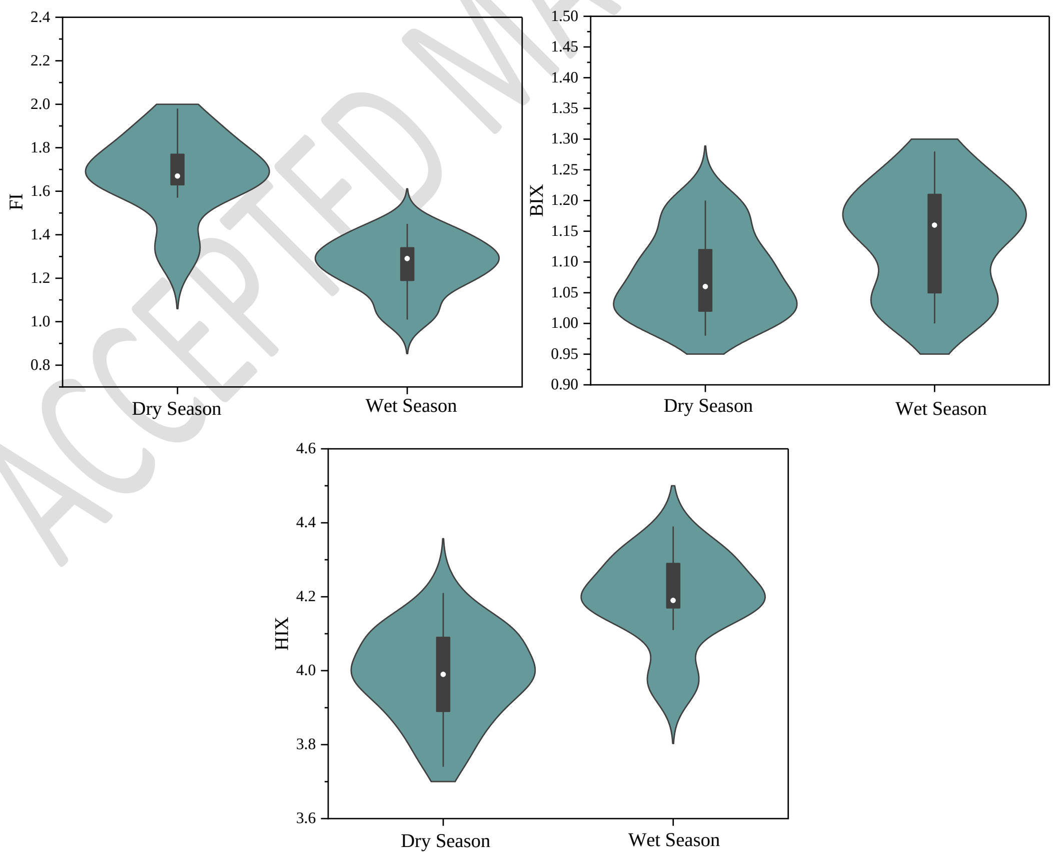

Figure 7. Properties ofthe mean of FI、BIX, HX in the wet season and the dry season

The biological index (BIX) measures the autochthonous characteristics of DOM in water bodies, which are proportional to each other (Chen et al. 2017). BIX value between 0.60 and 0.80 suggests minimal autochthonous contribution, the range of 0.8 to 1.0 indicates a more prominent autochthonous feature, and when BIX is greater than 1.00, DOM is primarily sourced from within the water body. As shown in Figure 5b, BIX of samples in the wet season period ranges from 1.00-1.27, with an average value of 1.13. The BIX of the samples in dry season was 0.98-1.17, with an average value of 1.09. Both mean values were in the range of 1.07-1.15, indicating that DOM of Luoma Lake showed strong autogenetic characteristics in both water periods. The contribution ratio of autogenetic sources was similiar, which was consistent with the conclusion obtained by FI analysis above.

The HIX index is to evaluate the degree of humification of DOM(Chen et al. 2017), when $HIX<1.50$, it indicates that DOM is mainly derived from microorganisms, when HIX is 1.50-3.00, it has weak humus characteristics and obvious endogenous characteristics, while $HIX>3.00$ indicates strong humus characteristics. The HIX values of the samples were between 3.74 and 4.21 (mean 3.99) during the dry season, and HIX values were between 3.95 and 4.39 (mean 4.18) during the wet season. There was no significant difference between the two seasons, indicating that the humification degree of DOM in both the dry and rainy seasons was weak, mainly composed of autotrophic sources. The flood runoff was similar to lake water, resulting in no significant change in lake water humification.

Comparison with other lakes, for example, in Poyang Lake, the export of floodplain-derived organics and river-lake interaction led to a remarkable increase in terrestrial aromatic and humic-like DOM with high molecular weights and long turnover times(Bai et al., 2016) . Further comparison with other lakes, e.g Gehu Lake, Dianshan Lake, and Yangcheng Lake had lower DOM concentrations and higher relative molecular weights.This may be due to the favorable environment provided by longer water retention times for microbial activity and pollutant accumulation. Previous studies have shown agricultural fertilization and human wastewater can cause relatively strong signals of small molecular weight proteins in lakes. This indicates that the types of human activities in the watershed also have a significant impact on the DOM structure(Chen et al., 2020;Shi et al., 2021).

In summary, the fluorescence parameters FI, HIX and BIX all indicate that the DOM in Luoma Lake is influenced by both endogenous and exogenous sources, and is mainly endogenous, which has strong autogenetic characteristics. FI in wet season was significantly lower than that in dry season, and the concentration of humus and protein-like substances in wet season was higher than that in dry season, which was consistent with the results of fluorescence intensity. HIX and BIX showed no anisotropy between two periods, indicating that the humification degree, autogenetic contribution ratio and biological activity of Luoma Lake had no effect with hydrological season.

3.3 Correlation between DOM and water environmental factors

Correlation analysis showed that during the flood season, TOC and a_{254} in Luoma lakes were significantly positively correlated ($p<0.01$), TOC and a_{254} were significantly positively correlated with components C1 ($p<0.01$). BIX was significantly negatively correlated and positively correlated with components C1 and C2, respectively ($p<0.01$), while HIX was positively correlated with components C1 and C2, respectively ($p<0.01$). There is a significant positive correlation ($p<0.01$) between component C1 and component C2. During the dry season, TOC and a_{254} were significantly positively correlated with component C1 ($p<0.01$). BIX was significantly positively correlated with components C3 ($p<0.01$). HIX is significantly positively correlated with component C1 and C2 ($p<0.01$), while significantly negatively correlated with the protein like component ($p<0.01$). Component C3 showed significant negative and positive correlations with components C1 and C2, respectively ($p<0.01$), and wto humic acid components have similar sources.

Table 1 Pearson's correlation coefficient between TOC concentration and parameters of Absorption fluorescence spectrum in the wet season and the dry season

Time	Index	a_{254}	$SUVA_{254}$	S_R	FI	BIX	HIX	C1	C2	C3
Flood Season	**TOC**	0.71	0.22	-0.31	-0.12	-0.21	-0.16	0.75	0.79	0.18
	a_{254}		-0.13	0.72	-0.58	-0.64	0.68	0.92	0.39	-0.31
	$SUVA_{254}$			-0.31	0.56	0.43	0.53	0.72	0.38	-0.0
	S_R				0.84	0.90	-0.92	-0.71	0.09	0.74
	FI					0.76	-0.75	-0.51	0.25	0.59
	BIX						-0.91	-0.65	0.25	0.74

	HIX							0.68	0.21	0.84
	C1								0.54	-0.31
	C2									0.43
Dry Season	**TOC**	0.71	0.22	-0.31	-0.12	-0.21	-0.16	0.75	0.79	0.18
	a_{254}		-0.13	0.72	-0.58	-0.64	0.68	0.92	0.39	-0.31
	$SUVA_{254}$			-0.31	0.56	0.43	0.53	0.72	0.38	-0.0
	S_R				0.84	0.90	-0.92	-0.71	0.09	0.74
	FI					0.76	-0.75	-0.51	0.25	0.59
	BIX						-0.91	-0.65	0.25	0.74
	HIX							0.68	0.21	0.84
	C1								0.54	-0.31
	C2									0.43

4. Conclusion

The DOM composition, structure and sources in Luoma lake were explored using optical indicators and EEM-PARAFAC modeled components. (1)three fluorescent components of the DOM in the Luoma lake were identified, i.e., humus-like components C1, C2 and protein-like component C3. In different hydrological scenarios, especially during the wet season, the input of rivers plays a key role in the source composition of DOM in Luoma lake. Under different hydrological scenarios, the humic and protein like components of the Luoma lake are both input from terrestrial and endogenous production. During the wet season, humic components are mainly input from terrestrial sources, while protein like components are mainly from endogenous sources. During the dry season, the contribution of protein-like components are more likely to originate from the autogenous sources, mainly from microorganisms or algae. The findings will be conducive to improving our understanding on the crucial role of DOM in biogeochemical cycles and provide key insights into water quality conservation in Luoma lake.

Competing interests

The authors have no competing interests to declare that are relevant to the content of this article.

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